ESRF	**Experiment title:** Electronic transitions in (Mg,Fe)SiO_3 perovskite and Fe-Ni alloys	**Experiment number**: HE-3174
Beamline: ID18	**Date of experiment**: from: 25.11.2009 to: 01.12.2009 (setup from 18.11.2009 to 24.11.2009)	**Date of report**: 26.02.2010
Shifts: 18	**Local contact(s)**: Dr. Aleksandr CHUMAKOV	*Received at ESRF:*

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Report:

The Earth's interior is not directly accessible; therefore we rely on seismology and its associated parameters (density, compressional and shear wave velocities) to provide information about its bulk properties. Equally important are laboratory measurements of the sound velocities of relevant minerals in order to extract information on parameters that cannot be measured using seismology, such as temperature and composition, which strongly influence the dynamics of the Earth's interior. The aim of experiment HE-3174 was to carry out nuclear inelastic scattering (NIS) measurements to determine sound velocities on lower mantle minerals at high pressure and temperature, combined with nuclear forward scattering (NFS) to determine their electronic structure.

Experiment HE3174 took place during operation in 7/8+1 mode (setup) and hybrid mode (25.11.09-01.12.09). For all measurements the beam was focussed to ca. 4 µm × 20 µm using a K-B mirror. A portable laser system was set up with a diamond anvil cell as described in Dubrovinsky et al. (2009), and the X-ray beam position relative to the location of the laser-heated spot was monitored during the experiment using a video camera.

In our previous experiment on ID18 involving laser heating in the diamond anvil cell (HE-2893 and HE-2901, which were carried out concurrently), we collected NIS data with the gasket horizontal to the beam (i.e., both the beam and the incoherent signal passed through the gasket) (Fig. 1, left). Although data collection was successful, there were two main problems associated with this setup: (1) the beam excites resonance in iron contained in the Be gasket; and (2) the beam excites resonance in parts of the sample that were not heated. For the present experiment we redesigned the laser heating system so that NIS data could be collected in a vertical geometry (Fig. 1, right). This new geometry solved both of the problems previously encountered.

Data collection commenced with $Fe_{0.2}Mg_{0.8}O$, the second most abundant lower mantle phase. We collected thirteen NIS spectra at pressures from 0 to 113 GPa at temperatures from ambient to 2200 K. Each NIS spectrum took roughly four to six hours to collect. Laser heating was monitored via the video camera during the NIS data collection and found to be stable. We also collected 27 NFS spectra of $Fe_{0.2}Mg_{0.8}O$ at the same conditions as the NIS data collection. A sample of $Fe_{0.12}Mg_{0.88}SiO_3$ perovskite was also studied using the same technique of laser heating in the diamond anvil cell. Data collection times were longer due to the smaller amount of iron in the sample, and the quality of the data was severely compromised by problems with beam purity (spurious bunches). For this sample we were only able to collect 2 NIS spectra at 50 GPa and 300 and 1100 K, and 4 NFS spectra at the same conditions. In addition, we collected 3 NFS spectra of Fe_3O_4 and Fe_2O_3 above 70 GPa, mainly for testing and optimisation of the system, and one NIS spectrum of $(NH_4)_2Mg^{57}Fe(CN)_6$ for energy calibration.

The NIS data for $Fe_{0.2}Mg_{0.8}O$ clearly show the influence of temperature through the increased intensity of the anti-Stokes part of the spectrum (Figs. 2 and 3). NFS spectra show the change in Fe^{2+} from high-spin (Fig. 2, top right) to low-spin (Fig. 2, bottom right and Fig. 3, right) as a function of pressure, and suggest a negative transition boundary at 40 GPa in contrast to existing models (Lin et al., 2007). The sound velocities extracted from the NIS data according to established procedures (Chumakov et al., 1996) show a discontinuity according to the influence of the spin transition (Lin et al., 2007), and for the first time we have been able to determine sound velocities for a lower mantle mineral at high pressure and high temperature. Analysis is continuing to enable comparison with seismology data, and preliminary results show a surprising behaviour. The single pressure at which we were able to study the effect of temperature on the sound velocity of the Earth's most abundant phase, $Fe_{0.12}Mg_{0.88}SiO_3$ perovskite, also shows a similar behaviour, but more data points are needed to confirm our unexpected observation.

References:

Chumakov, A.I. *et al.*, *Phys. Rev. B* **54**, R9596-R9599 (1996).
Dubrovinsky, L. *et al.*, *Journal of Synchrotron Radiation* **16**, 737-741 (2009).
Lin, J.-F. *et al.*, *Science* **317**, 1740-1743 (2007).

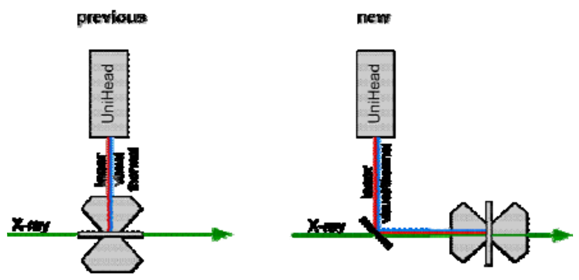

Fig. 1. Modified geometry for laser heating in the diamond anvil cell showing the path of X-rays (green) and the laser (red).

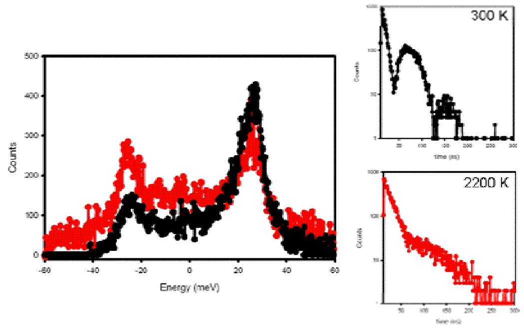

Fig. 2. $Fe_{0.2}Mg_{0.8}O$ at 40 GPa and 300 K (black) and 2200 K (red) showing data for NIS (left) and NFS (right).

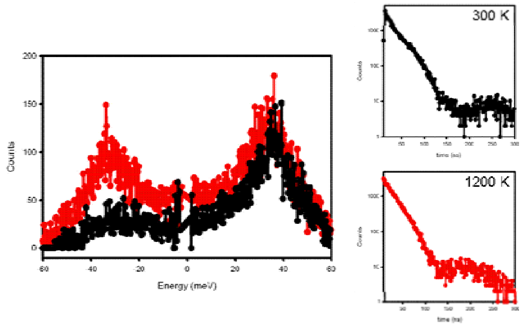

Fig. 3. $Fe_{0.2}Mg_{0.8}O$ at 101 GPa and 300 K (black) and 1200 K (red) showing data for NIS (left) and NFS (right).

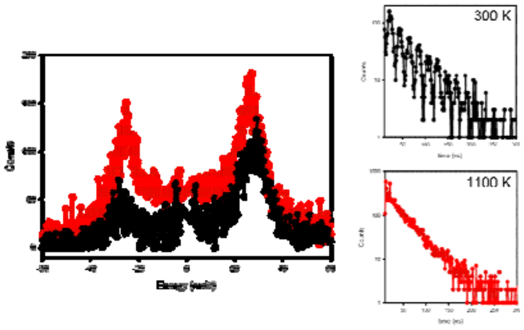

Fig. 4. $Fe_{0.12}Mg_{0.88}SiO_3$ pv at 50 GPa and 300 K (black) and 1100 K (red) showing data for NIS (left) and NFS (right).